

## PATENT ABSTRACTS OF JAPAN

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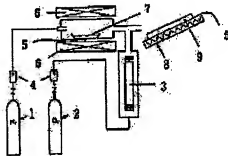
(72)Inventor : MARUYAMA TOSHIRO

## (54) METHOD FOR CHEMICAL VAPOR GROWTH OF SILICON DIOXIDE FILM

## (57)Abstract:

PURPOSE: To grow an  $\text{SiO}_2$  film by using a tetrakisdimethylaminosilane as an Si source, adding an oxygen source to the Si source and forming the film on the surface of a material to be treated.

CONSTITUTION: An Si source raw material such as a tetrakisdimethylaminosilane is vaporized in a vaporizer 7, heated and kept at a prescribed temperature with an electric furnace 6 and transported into a nozzle with a carrier gas such as  $\text{N}_2$  gas at a prescribed flow rate fed from a carrier gas cylinder 1. On the other hand, oxygen gas fed from an oxygen gas cylinder 2 is passed through an ozonizer 3, converted into an ozone gas-containing oxygen gas, fed into the nozzle and mixed with the Si source raw material. The obtained mixed gas is subsequently blown on a substrate (a material to be treated) 9 such as borosilicate glass heated at a low temperature of about  $40^\circ\text{C}$  with a heater 8. Thereby, the objective transparent film such as  $\text{SiO}_2$  good in adhesion is grown.



## LEGAL STATUS

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the manufacture approach of the film of the silicon dioxide film in more detail about the chemical-vapor-deposition approach.

[0002]

[Description of the Prior Art] chemical vapor deposition (Chemical Vapor Deposition, CVD) -- law is widely put in practical use as an approach of mass-producing the high performance film. The silane and the tetra-ethoxy silane (TEOS) are used as a source of silicon of the chemical-vapor-deposition method of the silicon dioxide film.

[0003]

[Problem(s) to be Solved by the Invention] However, a silane ignites in air and it is easy to hydrolyze a tetra-ethoxy silane with the moisture in air. Moreover, a tetra-ethoxy silane-ozone system cannot lower substrate temperature to 100 degrees C or less above 400 degrees C by the silane-oxygen system, either. This invention is made in view of the above-mentioned difficulty, and the place made into the purpose is in offer of the new chemical-vapor-deposition method which can compound the film of a silicon dioxide easily at the low substrate temperature near a room temperature using the raw material which is easy to deal with it.

[0004]

[Means for Solving the Problem] The chemical-vapor-deposition approach characterized by growing up the film of a silicon dioxide into processed material front faces, such as glass, a metal, and plastics, at the low substrate temperature near a room temperature by adding ozone to the source gas of silicon as a source of oxygen, using a tetrakis dimethylamino silane as a source of silicon.

[0005]

[Function] When based on this invention approach, using a raw material also with a safe handling top, it is few processes, and is low temperature, therefore the film of a silicon dioxide can be easily grown up into an including [ plastics ] processed material front face.

[Example] Hereafter, the example of this invention is explained. The mimetic diagram of membrane formation equipment is shown in drawing 1 R> 1. It evaporated by (7) in the carburetor which kept this at 40 degrees C, using a tetrakis dimethylamino silane as a source of silicon, and conveyed to the nozzle by making nitrogen gas (flow rate of 300cm<sup>3</sup>/min) (1) into carrier gas. It mixed with the oxygen gas (flow rate of 100cm<sup>3</sup>/min) containing the ozone as a source of oxygen in the nozzle, mixed gas was sprayed on the substrate (9), and membranes were formed under atmospheric pressure. Borosilicate glass was used as a substrate (9). When it was made to react at the substrate temperature of 40 degrees C, the transparent membrane of the good silicon dioxide of adhesion to a substrate was obtained by membrane formation rate 10 nm/min. The example of the infrared transparency spectrum of the thin film compounded by drawing 2 at four kinds of substrate temperature is shown. Moreover, when the oxygen gas (flow rate of 100cm<sup>3</sup>/min) which does not contain ozone as a source of oxygen was used, the transparent membrane of the good silicon dioxide [ as opposed to a substrate at the substrate temperature of 300 degrees C or more ] of adhesion was obtained by membrane formation rate 5 nm/min. The example of the infrared transparency spectrum of thin \*\* compounded by drawing 3 at two kinds of substrate temperature is shown. although the suitable example was given per this invention above and

many things were explained, this invention is not limited to this example and comes out not to mention the ability to change many within limits which do not deviate from the pneumonia of invention.

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CLAIMS

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[Claim(s)]

[Claim 1] The chemical-vapor-deposition approach characterized by growing up the film of a silicon dioxide into a processed material front face by adding the source of oxygen to the source of silicon, using a tetrakis dimethylamino silane as a source of silicon.

[Claim 2] The chemical-vapor-deposition approach given in the 1st term of a claim that the source of oxygen is ozone.

[Claim 3] The chemical-vapor-deposition approach given in the 1st term of a claim that the source of oxygen is oxygen gas.

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is the schematic diagram of equipment.

[Drawing 2] It is drawing showing the example of the infrared transparency spectrum of the film compounded at four kinds of substrate temperature, using ozone as a source of oxygen.

[Drawing 3] It is drawing showing the example of the infrared transparency spectrum of the film compounded at two kinds of substrate temperature, using oxygen gas as a source of oxygen.

[Description of Notations]

- 1 Carrier Chemical Cylinder
- 2 Oxygen Chemical Cylinder
- 3 Ozonator
- 4 Quantity-of-Gas-Flow Controller
- 5 Thermometric Element
- 6 Electric Furnace
- 7 Source Raw Material of Silicon
- 8 Heater
- 9 Substrate (Processed Material)

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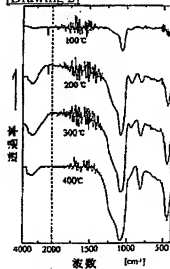
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## DRAWINGS

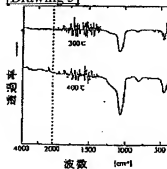
[Drawing 1]



[Drawing 2]



[Drawing 3]



[Translation done.]

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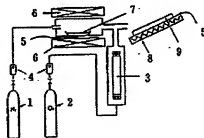
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(54)【発明の名称】 二酸化珪素膜の化学気相成長法

(57)【要約】

【目的】 取り扱いやすい原料を用いて、室温に近い低い基板温度で、二酸化珪素の膜を容易に合成し得る新たな化学気相成長法を提供する。

【構成】 テトラキスジメチルアミノシランをシリコン源として用い、これに酸素源としてのオゾンあるいは酸素ガスを加えることにより、被処理物表面に二酸化珪素の膜を成長させることを特徴とする化学気相成長方法による。





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## 【特許請求の範囲】

【請求項1】 テトラキスジメチルアミノシランをシリコン源として用い、シリコン源に酸素源を加えることにより、被処理物表面に二酸化珪素の膜を成長させることを特徴とする化学気相成長方法。

【請求項2】 酸素源がオゾンである特許請求範囲第1項記載の化学気相成長方法。

【請求項3】 酸素源が酸素ガスである特許請求範囲第1項記載の化学気相成長方法。

## 【発明の詳細な説明】

【0001】

【産業上の利用分野】 本発明は、化学気相成長方法に関し、さらに詳しくは、二酸化珪素膜の膜の製造方法に関する。

【0002】

【従来の技術】 化学気相成長 (Chemical Vapor Deposition, CVD) 法は、高性能膜を量産できる方法として広く実用化されている。二酸化珪素膜の化学気相成長法のシリコン源としてシランやテトラエトキシシラン (TEOS) が用いられている。

【0003】

【発明が解決しようとする課題】 しかし、シランは空気中で発火し、テトラエトキシシランは空気中の水分によって加水分解しやすい。また、基板温度もシラン-酸素系で400℃以上で、テトラエトキシシラン-オゾン系でも100℃以下に下げることができない。本発明は上記諸点に鑑みてなされたものであり、その目的とするところは、取り扱いやすい原料を用いて、室温に近い低い基板温度で、二酸化珪素の膜を容易に合成し得る新たな化学気相成長法の提供にある。

【0004】

【課題を解決するための手段】 テトラキスジメチルアミノシランをシリコン源として用い、酸素源として、例えば、オゾンを用いるシリコン源に加えることにより、室温に近い低い基板温度で、ガラス、金属、プラスチックなどの被処理物表面に二酸化珪素の膜を成長させることを特徴とする化学気相成長方法。

【0005】

【作用】 本発明方法によるときは、取り扱い上も安全な原料を用いて、少ない工程で、低温で、したがって、プラスチックを含む被処理物表面に、二酸化珪素の膜を

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容易に成長させることができる。

【実施例】 以下、本発明の実施例について説明する。図1に成膜装置の模式図を示す。シリコン源としてテトラキスジメチルアミノシランを用い、これを40℃に保った酸化器内(7)で酸化し、窒素ガス(流量300cm<sup>3</sup>/min)(1)をキャリアガスとしてノズルに輸送した。ノズル中で、酸素源としてのオゾンを含んだ酸素ガス(流量100cm<sup>3</sup>/min)と混合し、混合ガスを基板(9)上に吹き付け大気圧下で成膜した。基板(9)として、硼珪酸ガラスを用いた。基板温度40℃で反応させたところ、基板に対する密着性の良い二酸化珪素の透明膜が成膜速度10nm/minで得られた。図2に4種類の基板温度で合成された薄膜の赤外線透過スペクトルの例を示す。また、酸素源としてオゾンを含まない酸素ガス(流量100cm<sup>3</sup>/min)を用いた場合、基板温度300℃以上で、基板に対する密着性の良い二酸化珪素の透明膜が成膜速度5nm/minで得られた。図3に2種類の基板温度で合成された薄膜の赤外線透過スペクトルの例を示す。以上本発明につき好適な実施例を挙げて種々説明したが、本発明はこの実施例に限定されるものではなく、発明の精神を逸脱しない範囲内で多くの改変を施し得るものももちろんのことである。

## 【図面の簡単な説明】

【図1】 装置の概略図である。

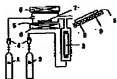
【図2】 酸素源としてオゾンを用いて4種類の基板温度で合成された膜の赤外線透過スペクトルの例を示す図である。

【図3】 酸素源として酸素ガスを用いて2種類の基板温度で合成された膜の赤外線透過スペクトルの例を示す図である。

## 【符号の説明】

- 1 キャリアガスボンベ
- 2 酸素ガスボンベ
- 3 オゾン発生器
- 4 ガス流量制御器
- 5 温度検出器
- 6 電気炉
- 7 シリコン源原料
- 8 ヒーター
- 9 基板(被処理物)

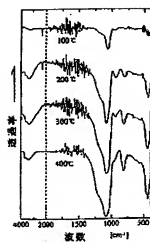
【図1】



(3)

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【図2】



【図3】

